CASE GS -- DPX-Y6202 STUDY 4 PM --

CHEM 128201 DPX-Y6202

BRANCH EAB DISC --

FORMULATION 00 - ACTIVE INGREDIENT

FICHE/MASTER ID No MRID CONTENT CAT 01

Cadwgan, G.E. and B. Atkins. 1985. Field soil dissipation [phenyl-^{14}C(U)] and [quinoxaline-^{14}C]DPX-Y6202 in Delaware, North Carolina, Illinois and Mississippi. Report No. AMR-333-85. Unpublished study prepared and submitted by E. I. du Pont de Nemours and Co., Inc., Wilmington, DE. Acc. No. 073771.

SUBST. CLASS = S.

DIRECT RVW TIME = 12 (MH) START-DATE END DATE

REVIEWED BY: L. Binari
TITLE: Staff Scientist
ORG: Dynamac Corp., Rockville, MD
TEL: 468-2500

APPROVED BY: J. Jordan
TITLE: Microbiologist
ORG: EAB/HED/OPP
TEL: 557-5457

SIGNATURE: J. Jordan DATE: 2/7/87

CONCLUSIONS:

Field Dissipation - Terrestrial

This study does not satisfy data requirements because an inappropriate method was used. Field dissipation data are required under actual use conditions. Also, the test subtance was not a typical end-use product, pretreatment soil samples were not collected and analyzed, field test data were incomplete and test material was not added to the soil at the highest label rate.

MATERIALS AND METHODS:

Stainless steel tubes (4 x 15 inches; inside diameter x length,) eight per site) were pushed 14.5 inches into untilled soil located in North Carolina, Mississippi, Delaware, and Illinois (Table 1). At each site the surface of the soil in the tubes was treated with quinoxaline-labeled [^{14}C]DPX-Y6202 (radiochemical purity 99.9%, specific activity 58 uCi/mg, Daiichi Chemical Co.) at 2.6-3.9 oz ai/A between May 16 and June 9, 1983.

At an additional site in DE (eight steel tubes), the soil was treated with phenyl-labeled [^{14}C]DPX-Y6202 (radiochemical purity 99.4%, specific activity 65 µCi/mg, Daiichi Chemical Co.) at 3.5 oz ai/A on July 21, 1983. Soil samples (the entire tube) were taken immediately after treatment and at 0.5, 1, 2, 4, 6, and 12 or 16 months posttreatment.

The cylinder of soil was frozen (-23°C), thawed for 1-2 days at room temperature, and dissected into segments (0-2, 2-4, 4-8 inches, and 8 inches-end). The soil segments were air-dried for ~1 day, milled, and frozen until analysis. Total radioactivity in the soil was quantified by combustion and LSC. Soil segments containing >5% of the applied radioactivity were extracted five times with acetonitrile:water: 85% phosphoric acid (90:10:1) followed by two extractions with acetone: aqueous 0.1 M ammonium carbonate (9:1). Extracted soil samples which still contained >20% of the applied radioactivity (only MS soil samples) were further extracted with acetonitrile:water:85% phosphoric acid (90:10:5) with heating (50-60°C) and ultrasonication. This was followed by extraction with acetone:aqueous 0.1 M ammonium carbonate (9:1) and aqueous 0.1 M ammonium carbonate with heating and ultrasonication.

All acidic and basic extracts were filtered, concentrated, diluted with water, and the organic solvents were removed by evaporation. The remaining aqueous phase was adjusted to pH 1.0 with 85% phosphoric acid and extracted twice with ethyl acetate. The ethyl acetate extracts were filtered, combined, and evaporated to dryness. The residue was dissolved in methylene chloride:acetone (1:1) and analyzed by TLC on silica gel with development in toluene:acetone:methanol:acetic acid (150:60:12:1). Unlabeled standards were cochromatographed with the extracts. Following development, DPX-Y6202 and its degradates were visualized under UV light and by autoradiography. Radioactive areas were scraped and quantified by LSC. Unextractable radioactivity remaining in the soil was quantified by combustion and LSC.

REPORTED RESULTS:

Total rainfall during the field tests was 56.4 inches at Fayetteville, North Carolina, 61.9 inches at Stoneville, Mississippi, 49.0 inches at Rochelle, Illinois, and 48.3 and 66.0 inches at Newark, Delaware (Table 2).

During the 12- and 16-month test periods, the majority of the applied radioactivity remained in the upper 4 inches of the soil columns (Table 2). At the first sampling interval (immediately posttreatment), parent DPX-Y6202 comprised only 9.1-47.9% of the applied radioactivity, while DPX-Y6202 acid comprised 36.9-66.3% of the applied (Tables 3 and 4). At 2 weeks posttreatment parent DPX-Y6202 comprised <9% of the applied. At 12 months after treatment of the loamy sand and silt loam soils with quinoxaline-labeled [^{14}C]DPX-Y6202, <2% of the applied DPX-Y6202 remained and degradates detected included DPX-Y6202 acid (1.1-14.4% of the applied radioactivity), phenol 1 (0.9-5.8%), phenol 2 (0.8-2.3%), and hydroxy-phenol 2 (4.6-23.1%). At 16 months after treatment of a silt loam soil with phenyl-labeled [^{14}C]DPX-Y6202, <3% of the applied DPX-Y6202 remained and degradates detected included DPX-Y6202 acid (8.0% of the applied radioactivity), phenol 1 (0.9%), phenol 3 (0.4%), and phenol 4

(2.2%). At 12 and 16 months posttreatment, unextractable [^{1}C]residues comprised 12.5-21.1% of the applied radioactivity.

DISCUSSION:

1. The 2-3 days used to thaw, air-dry, and grind the soils prior to analysis allowed further degradation of the test substance to occur. This procedure was inappropriate considering the rapid degradation rate of the test substance. The registrant provided results indicating that the test substance did not degrade significantly during the extraction procedure; 90% of the applied parent DPX-Y6202 was recovered from silt loam soil that was extracted immediately after fortification with quinoxaline-labeled [^{14}C]DPX-Y6202.

2. The dissipation of DPX-Y6202 from field soils enclosed by cylinders may not be representative of dissipation under actual use conditions. However, cylinders may be the only method for assessing very low volume pesticides, e.g., an ounce per acre (Glean) pesticide.

   The duPont Co. was given permission by EAB to conduct cylinder studies instead of actual field dissiption studies for some pesticides,e.g., Glean (chlorsulfuron). Glean was tested in the field using cylinders, because only from one-sixth to two-thirds ounce is applied per acre. However, from 5 to 40 ounces of Assure (quizalofop) are applied per acre. Only those very low application rate pesticides may be tested using field cylinders.

3. The test substance was not a typical end-use product.

4. Pretreatment soil samples or controls from untreated areas were not collected and analyzed.

5. Soil and air temperature data and the depth of the water table were not reported.

6. The data from the Rochelle, Illinois, site are too variable to assess the dissipation of DPX-Y6202. Total [^{14}C]residues recovered declined to 50% of the applied by 1 month posttreatment, then increased to 89% of the applied at 2 months posttreatment, and remained between 70-83% of the applied throughout the remainder of the test period.

Table 1. Soil characteristics.

Location	Soil type	Sand	Silt	Clay	Organic matter	pH	CEC (meq/100 g)
		%					
Fayetteville, North Carolina	Norfolk loamy sand	78	18	4	1.2	5.9	3.1
Stoneville, Mississippi	Dundee silt loam	8	74	18	1.4	6.4	5.3
Newark, Delaware	Keyport silt loam	21	62	17	2.8	6.4	8.2
Rochelle, Illinois	Flanagan silt loam	12	69	19	6.7	6.7	37.8

Table 2. Distribution of radioactivity (% of applied) in loamy sand and silt loam soils treated with quinoxaline-labeled or phenyl-labeled [^{14}C]DPX-Y6202 at 2.6-3.9 oz ai/A between May and July, 1983.[a]

Location/soil type	Date of application & rate	Sampling interval (months)	Sampling depth (inches) 0-2	2-4	4-8	8-end[b]	Total [^{14}C] recovered	Cumulative precipitation (inches)
			Soil treated with quinoxaline-labeled [^{14}C]DPX-Y6202					
Fayetteville, NC	May 17, 1983	0	103.2	0.7	0.5	0.2	104.6	0.6
Norfolk loamy sand	2.6 oz ai/A	0.5	51.5	7.4	2.6	0.5	62.1	2.5
		1	49.2	10.5	6.4	0.9	67.0	6.1
		2	39.5	6.1	2.8	0.5	48.9	9.9
		4	33.3	5.7	3.5	0.4	42.8	16.4
		6	23.3	1.7	1.6	0.1	26.7	21.7
		12	26.8	3.6	4.1	0.8	35.4	56.4
Stoneville, MS	May 18, 1983	0	87.4	7.0	0.3	0.1	94.8	0.6
Dundee silt loam	3.9 oz ai/A	0.5	53.7	0.3	0.1	0.1	54.2	7.5
		1	49.2	2.2	0.3	0.8	52.5	10.8
		2	58.7	6.5	1.6	0.2	67.0	14.8
		4	53.1	5.0	5.1	0.1	63.3	18.5
		6	55.2	3.1	0.9	0.3	59.4	22.6
		12	51.8	3.1	0.6	0.1	55.6	61.9
Newark, DE	June 9, 1983	0	99.3	ND[c]	ND	ND	99.3	0.6
Keyport silt loam	3.7 oz ai/A	0.5	86.7	3.4	2.2	0.4	92.7	2.6
		1	70.8	5.7	3.3	0.3	80.1	3.6
		2	71.9	3.3	2.0	0.6	77.8	4.0
		4	64.1	1.8	1.1	1.1	68.1	9.1
		6	51.3	5.1	2.3	0.7	59.4	22.1
		12	50.4	4.0	2.5	1.6	58.5	48.3
Rochelle, IL	May 16, 1983	0	95.0	2.8	0.1	ND	97.9	0.6
Flanagan silt loam	3.7 oz ai/A	0.5	48.2	3.0	0.2	ND	51.4	2.3
		1	48.4	1.4	0.6	0.1	50.5	7.4
		2	68.1	19.9	0.6	0.1	88.7	18.0
		4	60.1	11.0	0.7	0.2	72.0	22.7
		6	79.4	2.0	0.9	0.2	82.5	31.1
		12	74.0	5.1	0.1	1.0	80.2	49.0
			Soil treated with phenyl-labeled [^{14}C]DPX-Y6202					
Newark, DE	July 21, 1983	0	88.4	ND	ND	0.1	88.5	0.6
Keyport silt loam	3.5 oz ai/A	0.5	75.7	ND	ND	ND	75.7	1.0
		1	65.2	1.3	0.1	ND	66.6	2.0
		2	59.1	1.4	0.3	ND	60.9	3.7
		4	50.4	1.1	0.5	ND	52.1	15.8
		6	59.1	0.7	0.6	1.6	62.0	27.3
		16	31.8	0.3	0.2	0.5	32.8	66.0

[a] The soil was contained within stainless steel tubes in the ground and then treated.

[b] The study author stated that the total length of each cylinder of soil varied from 11-14 inches depending on the amount of compaction.

[c] Not detected, the detection was not specified.

Table 3. DPX-Y6202 and its degradates (% of applied radioactivity) in loamy sand and silt loam soils treated with quinoxaline-labeled [^{14}C]DPX-Y6202 (purity >99%).[a]

Sampling interval (months)	DPX-Y6202	DPX-Y6202 acid	Phenol 1	Phenol 2	Hydroxy-Phenol 2	Unknown 1	Unknown 3	Polar degradates[b]	Unextractable [^{14}C]	Total [^{14}C] recovered
					Norfolk loamy sand soil in Fayetteville, NC, treated at 2.6 oz ai/A					
0	22.7	42.3	4.1	3.1	3.1	6.2	2.1	19.6	3.0	106.2
0.5	3.7	13.6	3.7	2.4	12.6	3.7	2.1	6.0	10.7	58.5
1	3.1	4.8	2.5	1.3	14.7	5.2	3.2	8.3	19.5	62.6
2	2.8	2.6	1.8	1.0	12.4	3.4	2.3	4.3	17.8	48.4
4	0.8	1.9	0.6	1.0	8.1	2.6	2.6	4.1	18.6	40.3
6	0.2	1.0	0.6	0.8	4.8	1.8	1.4	2.2	11.8	24.6
12	0.7	1.1	1.0	1.1	4.6	2.2	1.9	1.9	12.5	27.0
					Dundee silt loam soil in Stoneville, MS, treated at 3.9 oz ai/A					
0	9.1	64.6	2.8	1.9	3.0	5.0	2.8	4.6	4.7	98.6
0.5	1.4	26.2	2.7	1.4	2.3	5.9	1.8	3.7	7.5	52.6
1	1.2	17.7	3.0	2.0	2.8	5.5	1.6	7.0	11.6	52.4
2	1.7	14.4	2.5	0.9	4.1	7.0	3.4	5.0	22.0	61.1
4	0.9	7.4	1.5	1.0	13.3	4.9	2.6	2.4	27.1	61.0
6	1.2	6.2	1.5	0.8	10.0	3.8	2.5	2.8	21.7	50.7
12	1.0	7.0	1.5	1.0	10.2	3.5	2.7	2.0	21.1	49.9
					Keyport silt loam soil in Newark, DE, treated at 3.7 oz ai/A					
0	24.7	66.3	0.1	0.1	0.1	0.1	0.1	0.2	8.3	99.3
0.5	3.2	17.3	3.2	3.3	26.8	5.7	0.2	4.4	24.7	88.7
1	1.6	12.0	2.0	1.7	20.0	4.0	2.8	0.2	24.5	83.6
2	4.4	4.6	1.9	2.2	27.4	3.1	1.4	0.2	21.1	66.1
4	0.8	5.1	1.5	2.6	19.4	4.2	2.2	0.2	30.2	66.0
6	0.3	3.2	0.8	0.8	16.1	4.8	1.3	5.5	17.3	50.1
12	1.2	4.6	0.9	0.8	14.5	4.0	1.2	3.6	20.1	50.9
					Flanagan silt loam soil in Rochelle, IL, treated at 3.7 oz ai/A					
0	47.9	36.9	1.8	0.9	1.8	1.8	0.9	1.8	1.3	95.1
0.5	2.3	12.6	1.8	1.8	5.9	6.3	2.3	12.2	3.4	48.6
1	1.3	10.1	2.1	1.7	8.8	6.8	2.5	10.2	4.3	47.3
2	2.8	27.0	6.6	2.3	20.3	7.7	2.9	6.2	12.8	88.6
4	2.0	17.2	4.3	1.6	19.6	4.9	2.3	2.8	14.0	68.7
6	1.7	13.3	4.1	2.3	25.5	6.4	0.1	4.7	19.1	77.1
12	1.7	14.4	5.8	2.3	23.1	5.2	3.5	2.9	17.9	76.8

[a] Results represent analysis of soil segments that contained >5% of the applied radioactivity.

[b] Radioactivity remaining at the origin of the TLC plates.

Table 4. DPX-Y6202 and its degradates (% of applied radioactivity) following the application of phenyl-labeled [^{14}C]DPX-Y6202 (purity >99%) at 3.5 oz ai/A to Keyport silt loam soil located in Newark, DE.[a]

Sampling interval (months)	DPX-Y6202	DPX-Y6202 acid	Phenol 1	Phenol 3	Phenol 4	Unknown 1	Unknown 2	Polar degradates[b]	Unextractable [^{14}C]	Total [^{14}C] recovered
0	25.9	46.8	1.9	2.3	3.8	1.4	1.1	1.2	1.3	85.7
0.5	8.6	40.4	2.8	0.7	6.4	3.6	0.9	2.8	10.2	76.4
1	7.6	24.6	0.7	0.3	6.8	5.4	2.1	3.6	10.8	61.9
2	5.1	20.5	1.9	0.7	9.6	4.4	0.3	2.0	15.6	60.1
4	4.4	18.0	1.5	0.6	7.9	3.6	0.6	2.0	11.0	49.6
6	5.4	18.4	2.8	0.5	8.2	4.2	0.4	1.9	17.2	59.0
16	2.8	8.0	0.9	0.4	2.2	2.4	0.6	0.9	15.9	34.1

[a] Results represent analysis of soil segments that contained > 5% of the applied radioactivity.

[b] Radioactivity remaining at the origin of the TLC plates.